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U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

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INTERNATIONAL APPLICATION NO.

INTERNATIONAL FILING DATE

PRIORITY DATE CLAIMED

PCT/DK99/00286

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TITLE OF INVENTION

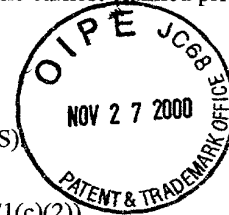
USE OF COLLOIDAL PRECIPITATED CALCIUM CARBONATE AS A FILTER IN THE PREPARATION OF PAPER

APPLICANT(S) FOR DO/EO/US

LUNDEN, Klaus A.; ATTRUP, Ib and MADSEN, Jens T.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau. WO 99/61703
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(3)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(2)).
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).



Items 11. to 16. below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.-1449 and International Search Report with cited references
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☒ Other items or information:
 - PCT/ISA/210
 - PCT/IPEA/409
 - PCT/RO/101
 - Two (2) sheets of formal drawings

PATENT
0459-0495P

IN THE U.S. PATENT AND TRADEMARK OFFICE

Applicant: LUNDEN, Klaus A. et al.
Int'l. Appl. No.: PCT/DK99/00286
Appl. No.: NEW Group:
Filed: November 27, 2000 Examiner:
For: USE OF COLLOIDAL PRECIPITATED
CALCIUM CARBONATE AS A FILTER IN
THE PREPARATION OF PAPER

PRELIMINARY AMENDMENT

BOX PATENT APPLICATION

Assistant Commissioner for Patents
Washington, DC 20231

November 27, 2000

Sir:

The following Preliminary Amendments and Remarks are respectfully submitted in connection with the above-identified application.

AMENDMENTS

IN THE SPECIFICATION:

Please amend the specification as follows:

Before line 1, insert --This application is the national phase under 35 U.S.C. § 371 of PCT International Application No. PCT/DK 99/00286 which has an International filing date of May 27, 1998, which designated the United States of America.--

IN THE CLAIMS:

Please cancel claims 1 to 23 without prejudice or disclaimer of the subject matter contained therein.

Please amend the claims as follows:

CLAIMS

24. A process for regulating the porosity and printing properties of uncoated wood-containing paper, the process comprising using a sufficient quantity of colloidal PCC having a BET surface area of 10-100 m²/g as a filler to achieve a desired porosity of the paper.
25. A process according to claim 24 wherein the paper is SC paper, in particular SC-A paper, and wherein colloidal PCC is used in a quantity sufficient to achieve a porosity of at most 0.30 μm/Pas.
26. A process according to claim 24 wherein the paper is SC-B paper, and wherein colloidal PCC is used in a quantity sufficient to achieve a porosity of at most 0.60 μm/Pas.
27. A process according to claim 24 wherein the paper is newsprint, and wherein colloidal PCC is used in an amount sufficient to achieve a porosity of at most 20 μm/Pas.
28. A process according to claim 24 wherein the colloidal PCC has a BET surface area of 15-50 m²/g.
29. A process according to claim 28 wherein the colloidal PCC has a BET surface area of 20-30 m²/g.
30. A process according to claim 24 wherein colloidal PCC is incorporated into the paper in an amount of at least about 1% by weight based on the total weight of the paper.
31. A process according to claim 30 wherein colloidal PCC is incorporated into the paper in an amount of at least about 2% by weight based on the total weight of the paper.
32. Uncoated wood-containing paper containing colloidal PCC.
33. Paper according to claim 32 containing colloidal PCC having a BET surface area of 10-100 m²/g as a filler.

31. Paper according to claim 33 comprising at least one further filler selected from the group consisting of non-colloidal PCC, kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

34. Paper according to claim 33 wherein the colloidal PCC has a BET surface area of 15-50 m²/g.

35. Paper according to claim 32 wherein the colloidal PCC is present in an amount of at least about 1% by weight based on the total weight of the paper.

36. SC paper containing colloidal PCC and having a porosity of at most 0.30 µm/Pas.

37. SC paper according to claim 36 wherein the paper is SC-A paper.

38. SC-B paper containing colloidal PCC and having a porosity of at most 0.60 µm/Pas.

39. Newsprint containing colloidal PCC and having a porosity of at most 20 µm/Pas.

40. Paper according to claim 36 comprising at least one further filler selected from the group consisting of non-colloidal PCC, kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

41. Paper according to claim 36 wherein the colloidal PCC has a BET surface area of 10-100 m²/g.

42. A pigment mixture suitable for paper manufacture and comprising colloidal PCC having a BET surface area of 10-100 m²/g in combination with at least one filler selected from the group consisting of : kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

43. A pigment mixture suitable for paper manufacture and comprising a combination of colloidal PCC having a BET surface area of 10-100 m²/g and non-colloidal PCC.

5

44. A pigment mixture according to any of claims 42-43 wherein the colloidal PCC comprises aggregates/agglomerates having an equivalent spherical particle size in the range 0.1-5.0 μm, wherein the aggregates/agglomerates consist of single crystals having
10 an equivalent spherical particle size of about 0.01-0.50 μm.

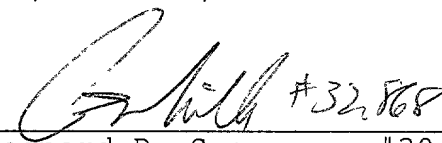
REMARKS

The specification has been amended to provide a cross-reference to the previously filed International Application. Also, the claims have been amended to delete multiple dependencies. Entry of the present amendment and favorable action on the above-identified application are respectfully requested.

If necessary, the Commissioner is hereby authorized in this, concurrent, and future replies, to charge payment or credit any overpayment to Deposit Account No. 02-2448 for any additional fees required under 37 C.F.R. § 1.16 or under 37 C.F.R. § 1.17; particularly, extension of time fees.

Respectfully submitted,

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(Rev. 04/19/2000)

USE OF COLLOIDAL PRECIPITATED CALCIUM CARBONATE AS A FILTER IN THE PREPARATION OF PAPER

FIELD OF THE INVENTION

- 5 The invention relates to use of colloidal PCC (precipitated calcium carbonate) as a filler in the preparation of paper for the purpose of controlling the porosity and printing properties of the paper.

BACKGROUND OF THE INVENTION

10

- In the connection with the manufacture of paper it is very important to be able to control the porosity of the paper. For example, a paper with low porosity is required in order to obtain an acceptable result in, e.g., ink-jet and rotogravure printing. If the paper is too porous it will function like blotting paper during printing and the resulting
- 15 print may appear blurred, the contrast between printed and unprinted areas or between differently coloured areas not being rendered sharply. Similarly, on a paper which is of non-uniform porosity it can be seen that the intensity of colouration varies ("mottling"), which is of course undesirable since the coloured surface appears variegated or mottled. On the other hand, the porosity of the paper can also be too
- 20 low, since a very dense paper will have difficulty in absorbing printing ink, which among other things may result in smudging ("set off") between printed sheets. This phenomenon can influence the printing results, the printing speed and the printing process employed in a negative manner.

- 25 The paper industry presently uses several different ways of regulating the porosity of paper. Use is made among other things of the fact that certain minerals in the form of flakes, e.g. talc and kaolin, will, as result of their form, be able to reduce the porosity since the individual particles will become deposited like the scales on a fish and thereby seal the surface. Fine silicates can be used in connection with pigmentation to
- 30 reduce the porosity of the paper. When they come into or onto the paper, these fine particles will close the pores which contribute to the porosity of the paper.

In order to regulate the properties of the paper, a combination of one or more fillers and a variety of other additives is often used. Among the group of additives are

alkylketene dimers (AKD), alkenylsuccinic acid anhydride (ASA), starch and retention agents. Retention agents are added to facilitate the manufacture of the paper, whilst AKD, ASA and starch are added to ensure the quality of the paper (strength, printing properties, etc.).

5

Regardless of which of the presently known methods is used, they all have drawbacks. Kaolin and talc in the form of flakes will negatively influence the brightness of the paper compared to the whiter fillers, such as ground marble or PCC (precipitated calcium carbonate).

10

The fine silicate products used for pigmentation have relatively good technical properties. However, the silicate products have the disadvantage of being much more expensive than the fillers normally used in paper manufacture. The same applies to other additives normally used in connection with paper manufacture. These are often
15 many times more expensive than a calcium carbonate filler.

Over the years, numerous attempts have been made to optimise paper compositions precisely for the purpose of improving the porosity and printing properties of the paper. The problem has been, however, that none of these approaches to a solution
20 have been ideal, since they have either had a negative influence on the other properties of the paper (among other things the brightness) or are relatively expensive to use (silicate products).

The use of colloidal PCC as such in paper is known. For example, US 4,892,590
25 discloses the use of a two-component binder system as a retention agent for paper manufacture, wherein the binder comprises colloidal PCC with a high specific surface area together with a cationic starch. The PCC used has a surface area of 10-200 m²/g, and the weight ratio between PCC and cationic starch is from 2:1 to 1:20.

30 US 4,460,637 discloses ink-jet paper (coated paper) with 2 different peaks of pore size distribution in the ink-receiving layer or layers. The desired pore size distribution can be achieved, inter alia by means of agglomerates with an average diameter of 1-50 µm in which the individual particles in the agglomerates have a size of at most

0.20 μm , e.g. colloidal particles of at most 0.01 μm ; such colloidal particles can be colloidal calcium carbonate.

It is not believed that colloidal PCC has previously been described or used as a filler in
5 paper for the purpose of controlling the porosity and printing properties of the paper.

DESCRIPTION OF THE INVENTION

It has now been found that use of colloidal PCC with a large surface area as a filler
10 makes it possible to replace a proportion of the previously mentioned pigments whilst also providing the possibility of regulating the porosity and printability properties of the paper. Compared with the previously described methods, the use of colloidal PCC has numerous advantages. It is cheap, produces low wear, it can produce greater
15 brightness than kaolin and talc flakes, and the product is more adaptable to individual types of paper.

In its broadest aspect, the present invention relates to the use of colloidal PCC as a filler to control the porosity and printing properties of paper, in particular to reduce the porosity relative to the porosity which can otherwise be achieved with other types of
20 fillers and pigments conventionally used in the manufacture of paper.

One aspect of the invention thus relates to a process for regulating the porosity and printing properties of paper, wherein a sufficient quantity of colloidal PCC having a BET surface area of 10-100 m^2/g is used as a filler to achieve a desired porosity of the
25 paper.

In another aspect, the invention relates to paper containing colloidal PCC as a filler.

In a third aspect, the invention relates to a pigment mixture which is suitable for
30 manufacture of paper and which contains colloidal PCC.

Other aspects and preferred embodiments will be apparent from the following detailed description of the invention.

As employed in the present description and claims, the term "colloidal PCC" (chemical formula: CaCO_3) designates a PCC product in the form of aggregates/agglomerates of individual PCC particles in which the aggregates/agglomerates have a surface area of at least $10 \text{ m}^2/\text{g}$ as determined by the BET method (Brunauer, Emmet, Teller, DIN 66131). The aggregates/agglomerates preferably have an equivalent spherical particle size (median particle size, MPS) in the range about $0.1\text{-}5.0 \mu\text{m}$, e.g. about $0.2\text{-}4 \mu\text{m}$, typically about $0.5\text{-}3.0 \mu\text{m}$, as determined e.g. by sedimentation on a Sedigraph 5100 from Micromeritics. The aggregates'/agglomerates' BET surface area will typically be up to about $100 \text{ m}^2/\text{g}$, more typically up to about $80 \text{ m}^2/\text{g}$, e.g. up to about $50 \text{ m}^2/\text{g}$, e.g. up to about $30 \text{ m}^2/\text{g}$ and typically at least about $15 \text{ m}^2/\text{g}$, e.g. at least about $20 \text{ m}^2/\text{g}$. The aggregates/agglomerates consist of a greater or smaller number of single crystals having an equivalent spherical particle size of, typically, about $0.01\text{-}0.50 \mu\text{m}$.

It will be apparent to the skilled person that colloidal PCC can also occur as aggregates with a surface area of less than $10 \text{ m}^2/\text{g}$, but as mentioned above the expression "colloidal PCC" in the context of the present application is to be understood as PCC with the stated surface area of at least $10 \text{ m}^2/\text{g}$. Correspondingly, according to the present invention a PCC mixture in which a part of the mixture is colloidal PCC with a surface area of at least $10 \text{ m}^2/\text{g}$ and a part of the mixture is "non-colloidal PCC" can be used, "non-colloidal PCC" being defined as PCC with a surface area of less than $10 \text{ m}^2/\text{g}$.

An example of a colloidal PCC product according to the invention is given in the table below:

25

Parameter	Value
Median particle size, MPS (μm)	1.5
Brightness ($R_{457}\text{-ISO, \%}$)	95.8
Surface area (BET, m^2/g)	25.0

The particle size distribution of this PCC product is shown in Fig. 1, whilst Fig. 2 shows a SEM picture of typical aggregates.

The colloidal PCC can, if desired, be used alone, i.e. as sole filler or pigment, in the manufacture of paper, but will presumably normally be used with at least one further

filler or pigment. These further fillers and pigments can be selected among both non-colloidal PCC and other types of fillers. There is a wide variety of types of PCC with different crystal forms which are suited as a filler, e.g. scalenohedral PCC, rhombohedral PCC, needle-shaped PCC (aragonite) and spherical PCC (vaterite).

- 5 Among other types of fillers and pigments which are suited for incorporation in paper, the following can be named: kaolin, calcined kaolin, talc, gypsum, ground marble, aluminium silicate, calcium silicate, magnesium silicate and other silicate-containing minerals, calcium sulphate, barium sulphate, titanium dioxide, zinc oxide, zinc carbonate, calcium sulfoaluminates (satin white), aluminium hydroxide, diatomaceous
- 10 earth, plastic particles and organic pigments. Paper manufactured according to the present invention can, in addition to the colloidal PCC, suitably contain one or more such PCC or non-PCC fillers or pigments to obtain the desired paper properties. Preferred further fillers are non-colloidal PCC, kaolin, calcined kaolin, talc, gypsum, chalk, ground marble, silicate-containing minerals and calcium sulfoaluminates. Non-
- 15 colloidal PCC, kaolin, calcined kaolin, chalk and ground marble are particularly preferred.

- The finding which forms the basis of the invention, namely the fact that the porosity of paper can be regulated accurately by means of colloidal PCC, provides the
- 20 advantage, however, that the relative amount of the colloidal PCC relative to other fillers and/or pigments, as well as the colloidal PCC's properties (especially the surface area), can be adjusted in each individual case in order to achieve the properties which are desired for the paper in question. It is thus clear that the amount of colloidal PCC which is to be used depends on the type of paper to be manufactured and on the type
- 25 and amount of any other fillers. The amount of colloidal PCC to be used can therefore vary widely, i.e. from about 1% by weight of the total filler up to 100% of the total filler. The colloidal PCC will normally be used in an amount of at least 10% by weight, more typically at least 20% by weight, e.g. at least about 50% by weight, based on the weight of the total filler. The precise amount of colloidal PCC to be used in order
- 30 to achieve the desired properties for a given paper, including a particular porosity, will be easily determined by the skilled person, e.g. by simply preparing a series of paper samples in which there are used different amounts of the colloidal PCC relative to the other fillers.

Typically, the amount of colloidal PCC used according to the invention will be at least about 1 % by weight based on the total weight of the paper, more typically at least about 2 % by weight, e.g. at least about 3 % by weight, such as at least about 4 % or 5 % by weight. Depending on the total amount of filler in the paper and the proportion of the filler that is comprised by the colloidal PCC, the colloidal PCC can of course be present in significantly higher amounts, however.

According to the invention, the colloidal PCC can be used as a filler to regulate the porosity and printing properties of any type of paper, including e.g. wood-containing paper such as super-calendered (SC) paper/newsprint and wood-free paper such as fine paper. The invention is particularly suited for regulating the porosity and printing properties of uncoated paper, more particularly uncoated wood-containing paper, since these properties can be difficult to regulate in such paper compared to coated paper, where the porosity is controlled by the coating layer. In a preferred embodiment, the invention relates to the use of the colloidal PCC in the preparation of SC paper.

It will be known to persons skilled in the art of paper manufacturing that the terms "wood-containing" and "wood-free" refer to whether or not the lignin component of the ligno-cellulose wood fibres has been removed. These terms are used herein in accordance with their conventional meanings in the art, i.e. "wood-free" refers to cellulose fibres in which substantially all or at least most of the lignin has been removed, whereas "wood-containing" refers to ligno-cellulose fibres in which the lignin component has not been removed. While the specific amount of lignin that can be present in "wood-free" pulp may vary from country to country, this amount is relatively small. For example, in Finland wood-free paper is defined as paper in which less than 10 % by weight of the pulp is groundwood or other lignin-containing pulp. In the present context, "wood-containing paper" thus refers to paper in which the fibres comprise a substantial lignin component, wherein typically at least about 5 % by weight of the pulp is lignin-containing pulp, more typically at least about 10 % by weight, such as at least about 15 or 20 % by weight.

Removal of lignin to result in wood-free fibres can be performed by means of various well-known processes, e.g. using the Kraft process or by sulphite pulping. Such

processes that remove lignin from the wood fibres result in higher quality, but also more expensive fibres.

In the case of SC paper, in particular SC-A paper, containing colloidal PCC according to the invention, the porosity can e.g. be reduced to a value of at most about 0.30 $\mu\text{m}/\text{Pas}$, e.g. at most about 0.28 $\mu\text{m}/\text{Pas}$, e.g. at most about 0.26 $\mu\text{m}/\text{Pas}$, e.g. at most about 0.24 $\mu\text{m}/\text{Pas}$, e.g. at most about 0.22 $\mu\text{m}/\text{Pas}$. In other words, the porosity of the paper can be reduced to a value around, or possibly even lower than, the value of the porosity of an equivalent paper prepared on the basis of kaolin; this is illustrated in Example 1.

The present invention also allows improved porosity values in SC-B paper. Thus, SC-B paper containing colloidal PCC according to the invention may have a porosity of at most about 0.60 $\mu\text{m}/\text{Pas}$, e.g. at most about 0.50 $\mu\text{m}/\text{Pas}$, e.g. at most about 0.40 $\mu\text{m}/\text{Pas}$, e.g. at most about 0.35 $\mu\text{m}/\text{Pas}$.

It will be known to persons skilled in the art that SC paper may be classified into one of several subcategories based on properties of brightness, filler level, roughness, sheet gloss and porosity. The top grade of SC paper is thus SC-A+. SC-A paper typically differs from SC-A+ in having a somewhat lower brightness, while SC-B typically differs from SC-A in having one or more of a lower brightness, a lower filler level, a lower sheet gloss and a higher porosity.

In the context of the present specification and claims, the SC paper grades SC-A, SC-A+ and SC-B are defined as follows.

SC-A

Brightness $\geq 64\%$

Filler level $\geq 30\%$

Roughness (0.5 bar) $\leq 2.0 \mu\text{m}$

Roughness (1 bar) $\leq 1.5 \mu\text{m}$

Porosity $\leq 0.3 \mu\text{m}/\text{Pas}$

SC-A+

As SC-A above, but brightness $\geq 70\%$

SC-B

- 5 SC papers that do not fulfil the requirements for SC-A, but which fulfil the following requirements:

Brightness $\geq 60\%$

Filler level $\geq 15\%$

Roughness (0.5 bar) $\leq 3.0 \mu\text{m}$

- 10 Roughness (1 bar) $\leq 2.5 \mu\text{m}$

Porosity $\leq 0.6 \mu\text{m/Pas}$

In the case of newsprint, the use of colloidal PCC according to the invention will make it possible to reduce the porosity of the paper to a value of at most about $20 \mu\text{m/Pas}$,

- 15 e.g. at most about $18 \mu\text{m/Pas}$, e.g. at most about $16 \mu\text{m/Pas}$; this is illustrated in Example 2. For SC paper, newsprint and other types of paper the porosity achieved in each case will depend among other things on the pulp used and on the amount and properties of the colloidal PCC and any other fillers used. The above mentioned porosity values for SC paper and newsprint, respectively, are therefore only to be
20 taken as examples, the important feature of the invention being the possibility of regulating (reducing) the porosity relative to the porosity which would otherwise be achievable in a given paper using a filler according to the prior art.

- Colloidal PCC can be prepared in a known manner by carbonating milk of lime (calcium
25 hydroxide slurry) under suitable conditions. The following conditions are to be regarded as a non-limiting example of the preparation of colloidal PCC:

- Burnt lime having a reactivity (DIN T_{60}) of between 10 sec. and 5 min. is slaked in
40°C warm water using a water/lime ratio of 4:1. The thus-prepared milk of lime is
30 diluted to 40% dry matter content, after which it is screened through a $500 \mu\text{m}$ screen.

After screening, the milk of lime is cooled to 20°C and carbonated in an appropriate gas flow reactor using flue gas or a CO₂-air mixture typically containing 20% CO₂. Carbonation is continued until the pH has fallen below 8.

- 5 At a gas flow of 2.5 m³ flue gas per m³ reactor volume the reaction will occur over a period of about 3 hours. After carbonation is completed the colloidal PCC is screened through a 45 µm screen.

The invention is further illustrated by the following non-limiting examples.

10

In the examples below, the following standards were used for determining paper properties:

Gram weight: Scan-P 6:75

- 15 Thickness: Scan-P 7:96

Density: Scan-P 7:96

Gloss: Tappi T480 om-92

Brightness: ISO 2470

Opacity: ISO 2471

- 20 Roughness: Scan-P 76:95

Porosity: PPS method

All amounts are by weight unless otherwise indicated.

25 EXAMPLE 1: REGULATION OF POROSITY IN SC PAPER

The following pigments were tested in SC paper:

	Kaolin reference Filler - M (ECC International)	Rhombohedral PCC Standard product (Faxo Paper Pigments A/S)	Colloidal PCC Experimental product (Faxo Paper Pigments A/S)
Brightness (R ₄₅₇ - ISO, %)	78.9	97.0	95.9
MPS (µm)	3.3	1.8	1.1
BET (m ² /g)	9.0	6.2	25

The test was carried out on a pilot paper machine with filler levels of 27, 30 and 33%.

- 5 The fibers were of Scandinavian origin and consisted of:

TMP (thermomechanical pulp) and GW (groundwood)	85%
Kraft (cellulose fibers processed by the "kraft" process)	15%

- 10 The following chemicals were used in the manufacturing process:

Retention agent	none
Other	none

pH adjusted to 7.3 by addition of H_3PO_4 .

15

For comparison purposes the results for paper are interpolated to 30% filler after calendering. The results are shown in the table below.

	Kaolin reference	Rhombohedral PCC	Colloidal PCC
Gram weight (g/m^2)	55	56	56
Thickness (μm)	49	54	55
Density (g/m^2)	1.123	1.030	1.020
Gloss (75° , %)	35	32	36
Brightness (R_{457} ISO, %)	69.6	76.3	72.5
Opacity (%)	86.8	90.0	85.9
Roughness (μm)	1.48	1.48	1.46
Porosity $\mu m/Pas$	0.19	0.32	0.21

- 20 It can be seen from the above table that colloidal PCC surprisingly is capable of lowering the porosity of the paper from 0.32 $\mu m/Pas$ using a standard PCC to 0.21 $\mu m/Pas$ with colloidal PCC, which is on a par with the kaolin reference.

EXAMPLE 2: REDUCTION OF POROSITY OF NEWSPRINT USING COLLOIDAL PCC AS

25 FILLER

The following pigments were tested in newsprint:

	Reference Calcined Kaolin (Ansilex from Engelhard)	Faxe Chalk 89 Chalk (Faxe Kridt A/S)	Rhombohedral PCC (Faxe Paper Pigments A/S)	Colloidal PCC Experimental product (Faxe Paper Pigments A/S)
Brightness (R ₄₅₇ -ISO, %)	89.6	87.4	96.2	95.7
MPS (μm)	0.9	1.5	1.2	1.1
BET (m ² /g)	15.0	3.2	9.2	23.0

The test was carried out on a pilot paper machine with filler levels from 2-10%.

5 The fibres consisted of:

Unbleached TMP (thermomechanical pulp)	95%
Bleached cellulose prepared by the sulphate process	5%

10 The following chemicals were used in the preparation:

Retention agent	Percol 230L (cationic polyacrylamide from Allied Colloids)
Other	none
pH adjusted to 7.3 by addition of H ₂ SO ₄ .	

15

For comparison purposes the results for paper are interpolated to 4% filler. The results are given in the following table, the gram weight of the papers being 46 g/m².

	Reference Calcined Kaolin (Ansilex from Engelhard)	Faxe Chalk 89 Chalk (Faxe Kridt A/S)	Rhombohedral PCC (Faxe Paper Pigments A/S)	Colloidal PCC Experimental product (Faxe Paper Pigments A/S)
Thickness (μm)	106	106	105	105
Roughness (μm)	5.2	6.2	6.2	6.2
Porosity (μm/Pas)	17	21	20	15
Brightness (R ₄₅₇ -ISO, %)	63.5	61.1	61.6	60.5
Opacity (%)	90.2	89.4	89.8	90.6

It can be seen from the table above that colloidal PCC surprisingly is able to lower the porosity of the paper from 21 $\mu\text{m}/\text{Pas}$ with a standard PCC to 15 $\mu\text{m}/\text{Pas}$ with colloidal PCC, which is lower than the kaolin reference at 4% filler level.

5 Conclusion

By using colloidal PCC as filler the porosity of the paper is lowered significantly. The amount of colloidal PCC in the paper can thereby be varied as required, so that the porosity and thereby also the printing properties can be regulated precisely. The

- 10 colloidal PCC can thus be used as required instead of or in combination with other conventional fillers and pigments in order to achieve the desired porosity.

EXAMPLE 3

- 15 A pigment mixture consisting of 50 parts (by weight) fine scalenohedral PCC, 30 parts fine rhombohedral PCC and 20 parts colloidal PCC was tested in production scale as a filler in SC-A grade paper at a commercial paper mill. The PCC pigment mixture was pH-stabilised by addition of a small amount of phosphoric acid in order to avoid the need for acid addition on the paper machine for pH-control. The properties of the PCC
- 20 mixture and the reference clay filler used in the trial are listed in the table below.

	Reference kaolin clay (European filler grade)	Experimental PCC mixture (Faxo Paper Pigments A/S)
Brightness (R_{457} -ISO %)	79.2	94.1
MPS (μm)	1.38	1.62
BET surface area (m^2/g)	11.7	10.8

The pulp furnish composition was 50 parts deinked pulp (DIP), 40-45 parts groundwood (GW) and 5-10 parts Kraft pulp.

25

The trial PCC mixture was tested at a constant total filler level with two levels of PCC addition. The balance to give the total amount of filler is reference clay and filler introduced with the DIP (recycled paper).

- 30 The properties of the papers resulting from the trial are listed in the table below.

	Reference	Trial 1	Trial 2
Added PCC ¹	0 %	10 %	20 %
Added clay ¹	32 %	22 %	12 %
Analysed CaCO ₃ content ¹	1.5 %	13.4 %	24.2 %
Analysed clay content ¹	37.6 %	25.6 %	15.2 %
Gram weight	57 g/m ²	56 g/m ²	56 g/m ²
Roughness TS (0.5 bar)	1.70 μ m	1.75 μ m	1.65 μ m
Roughness WS (0.5 bar)	1.70 μ m	1.70 μ m	1.55 μ m
Porosity (PPS) ²	0.122 μ m/Pa s	0.197 μ m/Pa s	0.228 μ m/Pa s
Gloss 75°, TS MD	50 %	45 %	45 %
Gloss 75°, WS MD	48 %	49 %	49 %
Brightness R ₄₅₇ -ISO	66.4 %	70.1 %	72.1 %
Opacity	92.1 %	92.5 %	91.8 %

1) By weight, based on the total weight of paper; TS = topside; WS = wireside; MD = machine direction

2) PPS = Parker-Print-Surf method

5

The runnability of the paper machine remained good during the two-day trial period and it was possible to increase the production capacity by 1.5%. The Hydrocol™ two-component retention system was used on the paper machine. The amount of cationic polymer could be reduced during the trial as the PCC pigment mixture was easier to retain than the reference clay. The pH in the paper machine headbox was 7.4 prior to the trial and it increased only slightly (to 7.6) during the trial.

The paper produced during the trial showed excellent results in full-scale commercial printing. It is remarkable that the paper brightness has been increased by 6 percentage points without any loss in opacity. The resulting 72% brightness is close to the superior SC-A+ quality.

EXAMPLE 4

20 A pigment mixture consisting of 80 parts (by weight) fine rhombohedral PCC and 20 parts colloidal PCC was tested in production scale as a filler in SC-B grade paper at a commercial paper mill. The rhombohedral PCC and the colloid PCC had BET surface areas of approximately 7 and 20 m²/g, respectively, to provide a mixture having an overall BET surface area of 9.1 m²/g as indicated below. The PCC pigment mixture

was pH-stabilised by addition of a small amount of phosphoric acid in order to avoid the need for acid addition on the paper machine for pH-control. The properties of the PCC mixture and the reference fillers used in the trial are listed in the table below.

Filler:	Reference kaolin clay (European filler grade)	Reference PCC	Experimental PCC mixture (Faxe Paper Pigments A/S)
Brightness (R_{457} -ISO %)	76.6	96.2	95.4
MPS (μm)	2.13	1.70	1.31
BET (m^2/g)	11.9	8.6	9.1

5

The pulp furnish composition was 30-35 parts deinked pulp (DIP), 10-15 parts chemothermomechanical pulp (CTMP) and groundwood (GW), adding up to a total of 100 parts.

- 10 The trial PCC mixture was tested at a constant total filler level with two levels of PCC addition. The balance to give the total amount of filler is reference clay and filler introduced with the DIP (recycled paper).

The properties of the papers resulting from the trial are listed in the table below.

15

	Reference	Trial 1	Trial 2
Added Faxe PCC ¹	0 %	11 %	18 %
Added reference PCC ¹	11 %	0 %	0 %
Added clay ¹	11 %	11 %	4 %
Analysed CaCO_3 content ¹	15.8 %	14.8 %	22.4 %
Analysed clay content ¹	17.8 %	17.4 %	11.2 %
Gram weight	57 g/m^2	56 g/m^2	56 g/m^2
Roughness TS (0.5 bar)	2.80 μm	2.90 μm	2.90 μm
Roughness WS (0.5 bar)	2.60 μm	2.80 μm	2.80 μm
Porosity (PPS)	0.570 $\mu\text{m}/\text{Pa s}$	0.514 $\mu\text{m}/\text{Pa s}$	0.554 $\mu\text{m}/\text{Pa s}$
Gloss 75°, TS MD	27 %	27 %	26 %
Gloss 75°, WS MD	24 %	25 %	24 %
Brightness R_{457} -ISO	63.4 %	62.6 %	64.8 %
Opacity	96.4 %	95.9 %	96.0 %

1) By weight, based on the total weight of paper; TS = topside; WS = wireside; MD = machine direction.

The runnability of the paper machine remained good during the two-day trial period and it was possible to increase the production capacity by 1.3%. The Hydrocol™ two-component retention system was used on the paper machine. The amount of cationic polymer could be reduced during the trial, as the PCC pigment mixture was easier to retain than the reference clay. The amount of blue and yellow colour could be reduced as well. The pH in the paper machine headbox was 7.3 prior to the trial and it was stable at 7.2 ± 0.1 during the trial.

The paper produced during the trial showed excellent results in full-scale commercial printing. The pulp bleaching was reduced in order to keep the paper brightness within the production specifications. The reduced amount of bleaching chemicals is an advantageous cost saving for the paper mill and environmentally beneficial.

EXAMPLE 5

A pigment mixture consisting of 80 parts (by weight) fine rhombohedral PCC and 20 parts colloidal PCC was tested in production scale as a filler in SC-A grade paper at a commercial paper mill. The PCC pigment mixture was pH-stabilised by addition of a small amount of phosphoric acid in order to avoid the need for acid addition on the paper machine for pH-control. The properties of the PCC mixture and the reference clay fillers used in the trial are listed in the table below. The paper mill alternates between use of two clays in their normal production.

Filler:	Reference kaolin clay (European filler grade)	Experimental PCC mixture (Faxo Paper Pigments A/S)
Brightness (R_{457} -ISO %)	80.7	94.1
MPS (μm)	1.79	1.62
BET (m^2/g)	15.4	10.8

The pulp furnish composition was 75 parts deinked pulp (DIP), 20 parts groundwood (GW) and 5 parts Kraft pulp.

The trial PCC mixture was tested at a constant total filler level with all fresh filler added being PCC. The balance to give the total amount of filler is filler introduced with the DIP (recycled paper). Paper was made in three gram weights: 48, 52 and 56 g/m². For the sake of simplicity only results for 56 g/m² are shown. The results at the other 5 gram weights were similar.

The properties of the papers resulting from the trial are listed in the table below.

	Reference	Trial
Added PCC ¹	0 %	17 %
Analysed CaCO ₃ content ¹	3.5 %	18.3 %
Analysed clay content ¹	32.9 %	17.4 %
Gram weight	57 g/m ²	57 g/m ²
Roughness TS (0.5 bar)	2.4 µm	2.20 µm
Roughness WS (0.5 bar)	2.65 µm	2.55 µm
Porosity (PPS)	0.252 µm/Pa·s	0.367 µm/Pa·s
Gloss 75°, TS MD	32.9 %	31.7 %
Gloss 75°, WS MD	26.1 %	29.2 %
Brightness R ₄₅₇ -ISO	66.0 %	66.4 %
Opacity	94.1 %	95.6 %

1) By weight based on the total weight of paper; TS = topside; WS = wireside; MD = machine direction.

The runnability of the paper machine remained good during the two-day trial period and it was possible to increase the production capacity by 1.2%. The Hydrocol™ two component retention system was used on the paper machine. The amount of cationic polymer could be reduced by approx. 20% during the trial as the PCC pigment mixture was easier retained than the reference clay. The pH in the paper machine headbox was 7.6 prior to the trial and it increased only slightly (to 7.7) during the trial.

The paper produced during the trial showed excellent results in full-scale commercial printing. It is remarkable that the paper mill had to totally stop bleaching their DIP in order to keep the brightness within the production specifications. This is a big economic advantage and also environmentally beneficial.

EXAMPLE 6

A number of fillers and filler mixtures were tested in a dynamic sheet former trial.

The fillers were three PCCs from Faxse Paper Pigments A/S, Denmark (a fine rhombohedral PCC, a fine scalenohedral PCC, and a colloidal PCC), and a kaolin clay from Dorfner. The properties of the fillers used in the trial are listed in the table below.

Filler	Brightness (R_{457} -ISO %)	MPS (μm)	BET (m^2/g)
Fine rhombohedral PCC	94.6	0.90	7.9
Fine scalenohedral PCC	95.7	2.13	9.3
Colloidal PCC	95.4	1.40	28.1
Kaolin clay (Dorfner)	81.7	2.02	8.4

5

Handsheets were made on a dynamic sheet former from Fibertech AB. The pulp furnish consisted of 50 parts groundwood, 30 parts DIP and 20 parts Kraft pulp. The target filler level was 35% by weight of the total weight of the paper. The results are listed below. The target gram weight of the handsheets was 56 g/m^2 (The actual gram weights varied between 53.2 and 58.1 g/m^2). Handsheets were made at three target filler levels, which were 30%, 33% and 36% filler by weight based on the total weight of the paper. The paper quality parameters were interpolated to a 35% filler level and the results are listed below.

10

Trial No:	1	2	3	4	5	6	7
Fine rhombohedral PCC ¹	100	80	70	50			
Fine scalenohedral PCC ¹					100	80	50
Colloidal PCC ¹		20	30	50		20	
Kaolin Clay ¹							50
Analysed CaCO_3 content ² (%)	32.5	32.9	32.3	32.7	32.9	32.8	18.8
Analysed clay content ² (%)	2.5	2.1	2.7	2.3	2.1	2.2	16.2
Gram weight (g/m^2)	54.2	53.9	55.0	56.1	57.0	56.5	56.9
Roughness TS (1 bar) (μm)	1.33	1.35	1.31	1.35	1.28	1.28	1.16
Porosity (PPS) ($\mu\text{m}/\text{Pa s}$)	0.276	0.271	0.265	0.252	0.337	0.259	0.236
Gloss 75° , TS MD (%)	32.0	35.1	37.2	40.1	39.7	40.2	46.7
Brightness R_{457} -ISO (%)	70.2	69.5	68.6	67.6	68.8	67.4	66.6
Opacity (%)	92.7	93.0	93.8	92.9	93.5	92.9	92.7

15 1) parts by weight, 2) By weight based on the total weight of paper; TS = topside; WS = wireside; MD = machine direction.

CLAIMS

1. A process for regulating the porosity and printing properties of uncoated paper, the process comprising using a sufficient quantity of colloidal PCC having a BET surface
5 area of 10-100 m²/g as a filler to achieve a desired porosity of the paper.
2. A process according to claim 1, wherein the paper is wood-containing paper.
3. A process according to claim 2, wherein the paper is SC paper, in particular SC-A
10 paper, and wherein colloidal PCC is used in a quantity sufficient to achieve a porosity of at most 0.30 µm/Pas, e.g. at most 0.28 µm/Pas, e.g. at most 0.26 µm/Pas, e.g. at most 0.24 µm/Pas, e.g. at most 0.22 µm/Pas.
4. A process according to claim 2, wherein the paper is SC-B paper, and wherein
15 colloidal PCC is used in a quantity sufficient to achieve a porosity of at most 0.60 µm/Pas, e.g. at most 0.50 µm/Pas, e.g. at most 0.40 µm/Pas, e.g. at most 0.35 µm/Pas.
5. A process according to claim 2, wherein the paper is newsprint, and wherein
20 colloidal PCC is used in an amount sufficient to achieve a porosity of at most 20 µm/Pas, e.g. at most 18 µm/Pas, e.g. at most 16 µm/Pas.
6. A process according to any of the preceding claims, wherein the colloidal PCC has a BET surface area of 15-50 m²/g.
25
7. A process according to claim 6, wherein the colloidal PCC has a BET surface area of 20-30 m²/g.
8. A process according to any of the preceding claims, wherein colloidal PCC is
30 incorporated into the paper in an amount of at least about 1 % by weight based on the total weight of the paper.
9. A process according to claim 8, wherein colloidal PCC is incorporated into the paper in an amount of at least about 2 % by weight based on the total weight of the paper.

10. Paper containing colloidal PCC having a BET surface area of 10-100 m²/g as a filler.

- 5 11. Paper according to claim 10, comprising at least one further filler selected from non-colloidal PCC, kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

10

12. Paper according to claim 10 or 11, wherein the colloidal PCC has a BET surface area of 15-50 m²/g, e.g. 20-30 m²/g.

13. Paper according to any of claims 10-12, said paper being uncoated.

15

14. Paper according to any of claims 10-13, wherein the colloid PCC is present in an amount of at least about 1% by weight, e.g. at least about 2% by weight, based on the total weight of the paper.

- 20 15. Uncoated wood-containing paper containing colloidal PCC.

16. SC paper containing colloidal PCC and having a porosity of at most 0.30 µm/Pas, e.g. at most 0.28 µm/Pas, e.g. at most 0.26 µm/Pas, e.g. at most 0.24 µm/Pas, e.g. at most 0.22 µm/Pas.

25

17. SC paper according to claim 16, wherein the paper is SC-A paper.

18. SC-B paper containing colloidal PCC and having a porosity of at most 0.60 µm/Pas, e.g. at most 0.50 µm/Pas, e.g. at most 0.40 µm/Pas, e.g. at most 0.35 µm/Pas.

30

19. Newsprint containing colloidal PCC and having a porosity of at most 20 µm/Pas, e.g. at most 18 µm/Pas, e.g. at most 16 µm/Pas.

20. Paper according to any of claims 15-19, comprising at least one further filler selected from non-colloidal PCC, kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

21. Paper according to any of claims 15-20, wherein the colloidal PCC has a BET surface area of 10-100 m²/g, e.g. 15-50 m²/g, e.g. 20-30 m²/g.

22. A pigment mixture suitable for paper manufacture and comprising colloidal PCC having a BET surface area of 10-100 m²/g in combination with at least one filler selected from the following pigments: kaolin, calcined kaolin, gypsum, chalk, ground marble, silicate-containing minerals, sulphate-containing minerals, oxide-containing minerals, carbonate-containing minerals, hydroxide-containing minerals, calcium sulfoaluminates, plastic particles and organic pigments.

23. A pigment mixture suitable for paper manufacture and comprising a combination of colloidal PCC having a BET surface area of 10-100 m²/g and non-colloidal PCC.

24. A pigment mixture according to claim 22 or 23, wherein the colloidal PCC has a BET surface area of 15-50 m²/g, e.g. 20-30 m²/g.

25. A pigment mixture according to any of claims 22-24, wherein the colloidal PCC comprises aggregates/agglomerates having an equivalent spherical particle size in the range 0.1-5.0 µm, e.g. 0.2-4 µm, typically 0.5-3.0 µm, wherein the aggregates/agglomerates consist of single crystals having an equivalent spherical particle size of about 0.01-0.50 µm.

1/2

+ Cumulative mass percent smaller vs. diameter
* Mass population vs. diameter

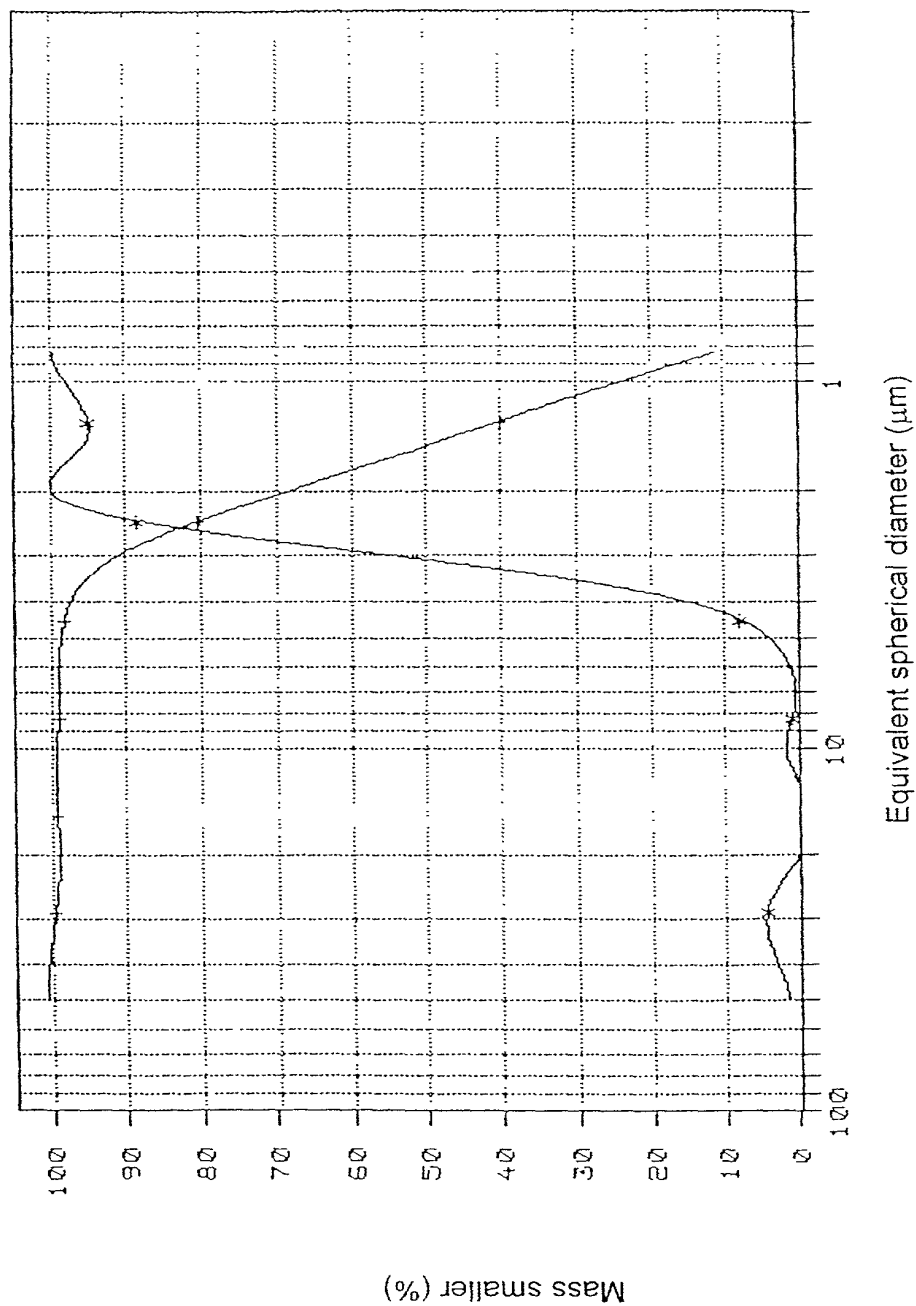


Fig. 1

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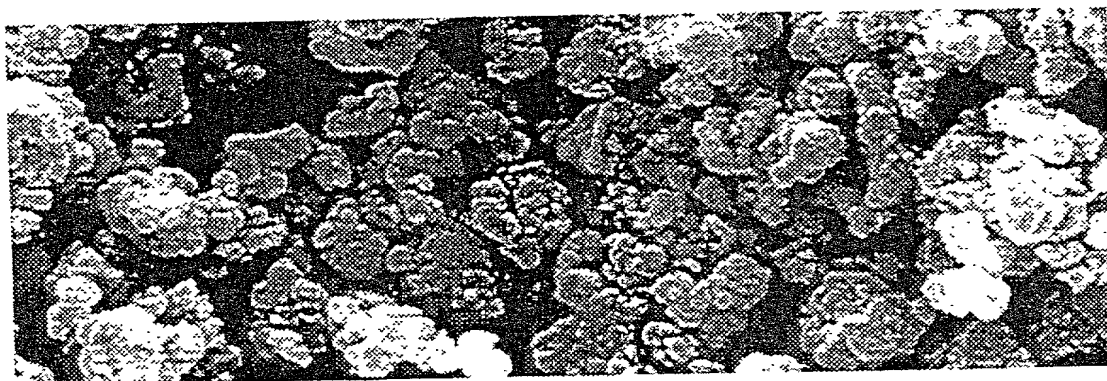


Fig. 2

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As a below named inventor, I hereby declare that: my residence, post office address and citizenship are as stated next to my name; that I verily believe that I am the original, first and sole inventor (if only one inventor is named below) or an original, first and joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

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USE OF COLLOIDAL PRECIPITATED CALCIUM CARBONATE AS A FILLER IN THE PREPARATION OF PAPERFill in Appropriate
Information -
For Use Without
Specification
Attached:

the specification of which is attached hereto. If not attached hereto,

the specification was filed on November 27, 2000 as
United States Application Number 09/701,261;
and amended on _____ (if applicable) and/or
the specification was filed on May 27, 1999 as PCT
International Application Number PCT/DK99/00286; and was
amended under PCT Article 19 on _____ (if applicable)

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I do not know and do not believe the same was ever known or used in the United States of America before my or our invention thereof, or patented or described in any printed publication in any country before my or our invention thereof or more than one year prior to this application, that the same was not in public use or on sale in the United States of America more than one year prior to this application, that the invention has not been patented or made the subject of an inventor's certificate issued before the date of this application in any country foreign to the United States of America on an application filed by me or my legal representative or assigns more than twelve months (six months for designs) prior to this application, and that no application for patent or inventor's certificate on this invention has been filed in any country foreign to the United States of America prior to this application by me or my legal representatives or assigns, except as follows.

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Prior Foreign Application(s)**Priority Claimed**Insert Priority
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(if appropriate)

<u>PA 1998 00735</u> (Number)	<u>DENMARK</u> (Country)	<u>May 27, 1998</u> (Month/Day/Year Filed)	<input checked="" type="checkbox"/> Yes	<input type="checkbox"/> No
_____ (Number)	_____ (Country)	_____ (Month/Day/Year Filed)	<input type="checkbox"/> Yes	<input type="checkbox"/> No
_____ (Number)	_____ (Country)	_____ (Month/Day/Year Filed)	<input type="checkbox"/> Yes	<input type="checkbox"/> No
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I hereby claim the benefit under Title 35, United States Code, §120 of any United States and/or PCT application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States and/or PCT application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose information which is material to the patentability as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

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(if any)

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I hereby appoint the following attorneys to prosecute this application and/or an international application based on this application and to transact all business in the Patent and Trademark Office connected therewith and in connection with the resulting patent based on instructions received from the entity who first sent the application papers to the attorneys identified below, unless the inventor(s) or assignee provides said attorneys with a written notice to the contrary:

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